

May 16, 2016

Tom Moe  
USS Corporation  
P.O. Box 417  
Mountain Iron, MN 55768


RE: Project: NPDES-TB Wk1  
Pace Project No.: 1265452

Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on May 05, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,



Melisa M Woods  
melisa.woods@pacelabs.com  
Project Manager

Enclosures

cc: Cory Hertling  
Terri Sabetti, NTS



## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
without the written consent of Pace Analytical Services, Inc..

## CERTIFICATIONS

Project: NPDES-TB Wk1

Pace Project No.: 1265452

---

### Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792

Alaska Certification #MN01084

Arizona Department of Health Certification #AZ0785

Minnesota Dept of Health Certification #: 027-137-445

North Dakota Certification: # R-203

Wisconsin DNR Certification # : 998027470

WA Department of Ecology Lab ID# C1007

Nevada DNR #MN010842015-1

Oklahoma Department of Environmental Quality

---

### Duluth Minnesota Certification ID's

4730 Oneota St., Duluth, MN 55807

Minnesota Dept of Health Certification #: 027-137-152

Wisconsin DNR Certification # : 999446800

North Dakota Certification #: R-105

---

## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
without the written consent of Pace Analytical Services, Inc..

## SAMPLE SUMMARY

Project: NPDES-TB Wk1

Pace Project No.: 1265452

Lab ID	Sample ID	Matrix	Date Collected	Date Received
1265452001	SD 001 (Seep 020)	Water	05/05/16 10:45	05/05/16 13:00

## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
without the written consent of Pace Analytical Services, Inc..

## SAMPLE ANALYTE COUNT

Project: NPDES-TB Wk1

Pace Project No.: 1265452

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
1265452001	SD 001 (Seep 020)	EPA 1664 TPH	BT1	1	PASI-DUL
		USGS I-3765	BEM	1	PASI-V
		EPA 300.0	DMB	1	PASI-V

## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
without the written consent of Pace Analytical Services, Inc..

## ANALYTICAL RESULTS

Project: NPDES-TB Wk1

Pace Project No.: 1265452

Sample: SD 001 (Seep 020)		Lab ID: 1265452001		Collected: 05/05/16 10:45		Received: 05/05/16 13:00		Matrix: Water	
Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>1664 SGT-HEM, TPH</b>									
Analytical Method: EPA 1664 TPH									
Total Petroleum Hydrocarbons	ND	mg/L	3.1	0.92	1		05/10/16 16:44		
<b>USGS I-3765 TSS</b>									
Analytical Method: USGS I-3765									
Total Suspended Solids	1.6	mg/L	1.0	1.0	1		05/10/16 13:08		
<b>300.0 IC Anions 28 Days</b>									
Analytical Method: EPA 300.0									
Sulfate	1130	mg/L	20.0	10.0	10		05/11/16 07:31	14808-79-8	

## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
without the written consent of Pace Analytical Services, Inc..

## QUALITY CONTROL DATA

Project: NPDES-TB Wk1

Pace Project No.: 1265452

QC Batch: DUL/6554

Analysis Method: EPA 1664 TPH

QC Batch Method: EPA 1664 TPH

Analysis Description: 1664 SGT-HEM, TPH

Associated Lab Samples: 1265452001

METHOD BLANK: 315410

Matrix: Water

Associated Lab Samples: 1265452001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Total Petroleum Hydrocarbons	mg/L	ND	3.0	0.90	05/10/16 14:35	

LABORATORY CONTROL SAMPLE: 315411

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Total Petroleum Hydrocarbons	mg/L	20	16.3	82	64-132	

MATRIX SPIKE SAMPLE: 315412

Parameter	Units	1265528001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Total Petroleum Hydrocarbons	mg/L	<1.1	23.3	17.4	71	64-132	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
without the written consent of Pace Analytical Services, Inc..

## QUALITY CONTROL DATA

Project: NPDES-TB Wk1

Pace Project No.: 1265452

QC Batch: WET/23931

Analysis Method: USGS I-3765

QC Batch Method: USGS I-3765

Analysis Description: USGS I-3765 Total Suspended Solids

Associated Lab Samples: 1265452001

METHOD BLANK: 315440

Matrix: Water

Associated Lab Samples: 1265452001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Total Suspended Solids	mg/L	ND	1.0	1.0	05/10/16 13:07	

LABORATORY CONTROL SAMPLE: 315441

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Total Suspended Solids	mg/L	239	238	100	80-120	

SAMPLE DUPLICATE: 315442

Parameter	Units	1265411001 Result	Dup Result	RPD	Max RPD	Qualifiers
Total Suspended Solids	mg/L	412	388	6	10	

SAMPLE DUPLICATE: 315443

Parameter	Units	1265412001 Result	Dup Result	RPD	Max RPD	Qualifiers
Total Suspended Solids	mg/L	32.0	32.0	0	10	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
without the written consent of Pace Analytical Services, Inc..

## QUALITY CONTROL DATA

Project: NPDES-TB Wk1

Pace Project No.: 1265452

QC Batch: WETA/16716

Analysis Method: EPA 300.0

QC Batch Method: EPA 300.0

Analysis Description: 300.0 IC Anions

Associated Lab Samples: 1265452001

METHOD BLANK: 315236

Matrix: Water

Associated Lab Samples: 1265452001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Sulfate	mg/L	ND	2.0	1.0	05/10/16 21:02	

LABORATORY CONTROL SAMPLE: 315237

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Sulfate	mg/L	50	49.1	98	90-110	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 315238 315239

Parameter	Units	1265372001 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Sulfate	mg/L	118	50	50	169	169	104	103	90-110	0	20	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 315240 315241

Parameter	Units	1265667001 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Sulfate	mg/L	49.6	50	50	102	102	104	104	90-110	0	20	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
without the written consent of Pace Analytical Services, Inc..



## QUALIFIERS

Project: NPDES-TB Wk1

Pace Project No.: 1265452

---

### DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

### LABORATORIES

PASI-DUL Pace Analytical Services - Duluth

PASI-V Pace Analytical Services - Virginia

## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
without the written consent of Pace Analytical Services, Inc..

## QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: NPDES-TB Wk1

Pace Project No.: 1265452

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
1265452001	SD 001 (Seep 020)	EPA 1664 TPH	DUL/6554		
1265452001	SD 001 (Seep 020)	USGS I-3765	WET/23931		
1265452001	SD 001 (Seep 020)	EPA 300.0	WETA/16716		

## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
without the written consent of Pace Analytical Services, Inc..

PM: MMW  
CLIENT: USS CORP  
Due Date: 05/19/16  
1203452

Due Date: 05/19/16


1

[illegible]

PM: MMW  
1203452  
CLIENT: USS CORP  
Due Date: 05/19/16

1

[illegible][illegible]

	Document Name: <b>Sample Condition Upon Receipt Form</b>	Document Revised: 23Feb2015 Page 1 of 1
	Document No.: <b>F-VM-C-001-Rev.09</b>	Issuing Authority: Pace Virginia, Minnesota Quality Office

**Sample Condition  
Upon Receipt**

Client Name:

Project #:

**WO# : 1265452**

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☒ Client  
☐ Commercial ☐ Pace ☐ Other: \_\_\_\_\_



Tracking Number: \_\_\_\_\_

Custody Seal on Cooler/Box Present? ☐ Yes ☒ No      Seals Intact? ☐ Yes ☒ No      Optional: Proj. Due Date: \_\_\_\_\_ Proj. Name: \_\_\_\_\_

Packing Material: ☐ Bubble Wrap ☐ Bubble Bags ☐ None ☒ Other: Unpacked      Temp Blank? ☒ Yes ☐ No

Thermometer Used: ☒ 140792808      Type of Ice: ☒ Wet ☐ Blue ☐ None ☒ Samples on ice, cooling process has begun

Cooler Temp Read °C: 5.1      Cooler Temp Corrected °C: 5.4      Biological Tissue Frozen? ☐ Yes ☐ No ☒ NA  
Temp should be above freezing to 6°C      Correction Factor: 10.3      Date and Initials of Person Examining Contents: 5/5/16

Comments:

Chain of Custody Present?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	1.
Chain of Custody Filled Out?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	2.
Chain of Custody Relinquished?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	3.
Sampler Name and Signature on COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	4.
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	5.
Short Hold Time Analysis (<72 hr)?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	6.
Rush Turn Around Time Requested?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	7.
Sufficient Volume?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	8.
Correct Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	9.
-Pace Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	
Containers Intact?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	10.
Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	12.
-Includes Date/Time/ID/Analysis Matrix: <u>wt</u>		
All containers needing acid/base preservation will be checked and documented in the pH logbook.	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	13.
Headspace in VOA Vials (>6mm)?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	14.
Trip Blank Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	15.
Trip Blank Custody Seals Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	
Pace Trip Blank Lot # (if purchased):		

**CLIENT NOTIFICATION/RESOLUTION**

Field Data Required? ☐ Yes ☐ No

Person Contacted: \_\_\_\_\_ Date/Time: \_\_\_\_\_

Comments/Resolution: \_\_\_\_\_

FECAL WAIVER ON FILE ☐ Y ☐ N

TEMPERATURE WAIVER ON FILE ☐ Y ☐ N

Project Manager Review:

Date: 5/5/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)

# Intra-Regional Chain of Custody

**Pace Analytical**  
www.pacelabs.com

Workorder: 1265452

Workorder Name: NPDES-TB Wk1

Owner Received Date: 5/5/2016

Due Date: 5/19/2016


Received at:  
Pace Analytical Virginia  
315 Chestnut Street  
Virginia, MN 55792  
Phone (218) 742-1042

Send To Lab:  
Pace Analytical Duluth  
4730 Oneota Street  
Duluth, MN 55807  
Phone (218) 727-6380

Report To:  
Melisa M Woods

Item	Sample ID	Sample Type	Collect Date/Time	Lab ID	Matrix	HCL	Preserved Containers	EPA 1664 TPH	Requested Analysis	LAB USE ONLY
1	SD 001 (Seep 020)	PS	5/5/2016 10:45	1265452001	Water	X				
2										
3										
4										
5										
Comments										
Transfers										
1	Released By	Date/Time	Received By	Date/Time						
2										
3										
4										
Cooler Temperature on Receipt 3.2°C										
Custody Seal (Y) or N										
Received on Ice (Y) or N										
Samples Intact (Y) or N										

\*\*\*In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document.  
This chain of custody is considered complete as is since this information is available in the owner laboratory.

	Document Name:	Document Revised: 22Jan2016
	Sample Condition Upon Receipt Form	Page 1 of 1
	Document No.: F-DUL-C-001-Rev.01	Issuing Authority: Pace Virginia, Minnesota Quality Office

**Sample Condition Upon Receipt**

Client Name:

Project #:

IR-COC

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client  
☐ Commercial ☐ Pace ☒ Other: Jack

Tracking Number:

Custody Seal on Cooler/Box Present? ☒ Yes ☐ No Seals Intact? ☒ Yes ☐ No

Optional: Proj. Due Date: Proj. Name:

Packing Material: ☐ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other: \_\_\_\_\_

Temp Blank? ☒ Yes ☐ No

Thermometer Used: ☒ B00051

Type of Ice: ☒ Wet ☐ Blue ☐ None ☒ Samples on ice, cooling process has begun

Cooler Temp Read °C: 2.0

Cooler Temp Corrected °C: 2.4

Biological Tissue Frozen? ☐ Yes ☐ No ☒ NA

Temp should be above freezing to 6°C

Correction Factor: +0.6°C

Date and Initials of Person Examining Contents: 5/5/16/cty

Comments:

Chain of Custody Present?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	1.
Chain of Custody Filled Out?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	2.
Chain of Custody Relinquished?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	3.
Sampler Name and Signature on COC?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	4.
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	5.
Short Hold Time Analysis (<72 hr)?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	6.
Rush Turn Around Time Requested?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	7.
Sufficient Volume?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	8.
Correct Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	9.
-Pace Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	
Containers Intact?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	10.
Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	12.
-Includes Date/Time/ID/Analysis Matrix: <u>WT</u>		
All containers needing acid/base preservation will be checked and documented in the pH logbook.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	13.
Headspace in VOA Vials (>6mm)?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	14.
Trip Blank Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	15.
Trip Blank Custody Seals Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	
Pace Trip Blank Lot # (if purchased):		

**CLIENT NOTIFICATION/RESOLUTION**

Field Data Required? ☐ Yes ☐ No

Person Contacted: \_\_\_\_\_

Date/Time: \_\_\_\_\_

Comments/Resolution: \_\_\_\_\_

FECAL WAIVER ON FILE Y N

TEMPERATURE WAIVER ON FILE Y N

Project Manager Review:

AP for LMF

Date: 5-6-16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)